Autchipillai Lakshmanan Pillai
Gowthami Thankachi Raghuvaran Jinu
Govindan Karthikeyan

https://doi.org/10.21278/TOF.41406
ISSN 1333-1124
eISSN 1849-1391

FABRICATION AND TRIBOLOGICAL BEHAVIOUR OF Mg-TiO_2 COMPOSITES

Summary

In the present study pure magnesium was taken as a base metal which is reinforced with various weight percentages of TiO_2 (2.5, 5, 7.5, 10 % wt). This is done through a vacuum stir casting route with argon as a shielding gas to prevent oxidation. The prepared samples were machined using the Electric Discharge Machining (EDM) to achieve accurate dimensions for the study of tribological properties. A computerized pin-on-disc machine is used to study the dry sliding wear behaviour of the Mg-based composites. Wear losses were calculated using a variety of load parameters (10N, 15N, 20N, and 25N), sliding velocities (1 m/s, 1.5 m/s, and 2.0 m/s) and the weight percentage of the reinforcement. The results proved that the wear loss increased with an increase in the load, and decreased with an increase in the sliding velocity. The wear resistance is improved with an increase in the percentage of reinforcements in the matrix. The morphology and distribution of reinforcements were analysed by means of Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray (EDX) spectroscopy, respectively. The results revealed a uniform distribution of TiO_2 particles throughout the magnesium matrix in the Mg-TiO_2 composites.

Key words: *magnesium matrix composites, titanium oxide, dry sliding wear test, SEM analysis*

1. Introduction

Magnesium-based composites are in great demand for weight-saving applications owing to their low density and good specific mechanical properties. The insatiable demand for energy savings has led to extensive research on lightweight materials as conventional materials are no longer able to satisfy the requirements of aerospace and automobile industries. In the integrated circuit industry, lightweight metals are used as replacements for plastics [1, 2]. Among the advantages of magnesium and its alloys used as composite matrices are excellent specific strength and stiffness, good damping capacities, and dimensional stability [3]. In spite of the attractive range of mechanical properties, relatively low strength, both at the ambient temperature and elevated temperatures, and poor resistance to wear and corrosion are a serious impediment to a wider application of magnesium alloys, comparable to the wide use of aluminium alloys. A further improvement in mechanical properties has been required to extend the application of magnesium alloys [3,4]. Several researchers reported that the addition of hard ceramic particles to magnesium resulted in better wear resistance and improved mechanical properties and thermal stability [5-6]. Magnesium is about by one third lighter than aluminium and by two thirds than titanium. Therefore, magnesium and titanium can be combined to get Mg-based titanium composites for structural applications. The intrinsic properties of titanium, such as hardness and ductility, and the good wettability of titanium and magnesium enhanced the strength and ductility of the Mg-Ti composite [7]. Simple casting technologies were found unsatisfactory for the fabrication of composites because of several structural defects, such as porosity, particle clustering, oxide inclusions, and interfacial reactions between the matrix and particles. Thus, a need for the stir casting process arose [8]. Stir casting is the most frequently used method for the production of particulate-reinforced cast metal matrix composites [9]. The primary processing followed by hot extrusion led to reduction in the porosity of composites and enhanced the interfacial bonding between the matrix and the reinforcement [10]. An addition of about 1% nano-sized titanium particles to magnesium improved the tensile and compressive strengths of composites [11-13]. Metal Matrix Composites (MMCs) exhibiting high wear resistance are ready to be used in the automobile and aerospace industries. A demand for good specific mechanical properties makes the research into the wear properties of Mg-MMCs of major importance [14]. Better wear resistance and micro hardness can be achieved by well-dispersed particles in the matrix [15]. Wear resistance can be further improved by the addition of nano-fillers to the Mg-matrix [16, 17]. Amicrostructural analysis indicates that the segregation of particles in the as-cast composite is largely eliminated by extrusion and that the particle distribution is drastically improved [18]. From the literature review one can conclude that magnesium composites, combined with the inherent advantages of the metal casting process, yield better cost-effective solutions to product needs in terms of part consolidation and weight savings than other materials and manufacturing methods. Several attempts have been made to investigate the effects of different types and sizes of reinforcements on the mechanical, the tribological and the microstructural behaviour of pure magnesium. Initially, micron-size reinforcements in the form of carbides (SiC, B_4C, ZrC, TiC), borides (ZrB_2, TiB_2), nitrides (TiN, ZrN, BN, AlN), and metals (Mo, Cu, Ti, Ni) were added to pure magnesium; subsequently, the observed effects were investigated [19-23]. In the available literature, one cannot find reports on attempts to study the effects of primary processing techniques, such as argon-controlled stir casting technique (liquid-phase synthesis) and powder metallurgy (PM) technique (solid-phase synthesis), on the microstructure and tribological properties of TiO_2-reinforced magnesium composites. This research paper is to elaborate a novel approach to the synthesis of TiO_2 and pure magnesium and the effect of TiO_2 reinforcements on tribological properties.

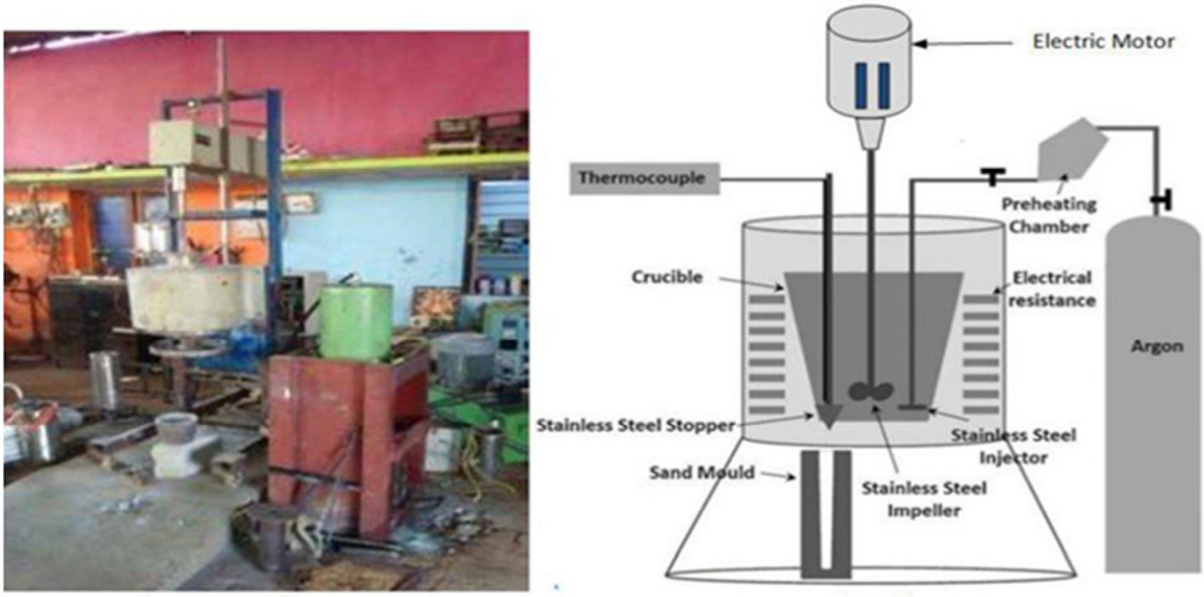
2. Materials and methods

2.1 Materials

Magnesium is the lightest metal among the metals available for structural applications. It is a silvery-white alkaline earth metal lighter than aluminium by one third. It burns in pure nitrogen and pure carbon dioxide. Magnesium reacts with cold water very slowly. It forms a thin protective coating of magnesium carbonate when it gets in touch with humid air.

The fire produced by magnesium cannot be extinguished by water since water reacts with hot magnesium and releases hydrogen which can cause the fire to burn more fiercely. Titanium is present in most igneous rocks and their sediments and it is always found bonded with another element that does not occur in natural pure form. Pure titanium is a silver-white transition metal resistant to corrosion (including both seawater and chlorine corrosion).

Titanium has the highest strength-to-weight ratio of all metals. Even though titanium is used in many products, nearly 95% of the purified metal is used to make titanium dioxide (TiO_2). The corrosion resistance of magnesium matrix can be improved by reinforcing it with TiO_2 particles which form a thin layer of TiO_2 on the surface of Mg-TiO_2composites. The TiO_2 layer acts as a corrosion protective layer.



(a) Photographic view (b) Schematic view

Fig. 1 (a-b) Stir casting setup with the argon shield

2.2 Stir casting method

The vacuum stir casting process is used to fabricate five samples (0%, 2.5%, 5%, 7.5%, and 10% wt of TiO_2 with pure magnesium). The experimental set up to produce the samples is presented in Figure 1(a-b).In the stir casting process, reinforcing phases usually in the powder form are distributed throughout the molten magnesium by means of mechanical stirring (with the use of an electric motor). The effect of high strength can be achieved by uniform distribution of secondary particles in the matrix, which is achieved by a stirring process. A mild steel impeller with a pitch of 45° is coupled with an electric motor through a shaft. The impeller is coated with zirconium alloys to prevent the iron contamination of molten metal. Since the magnesium alloy is highly sensitive to oxidation, there is a possibility of entrapment of gases and other inclusions during the stir casting process. Thus, the stirring process needs to be more tightly controlled with magnesium alloys than with aluminium alloys in order to prevent the entrapment of unwanted gases and other inclusions. Since magnesium is a flammable material and gets easily oxidized in the presence of oxygen, a shielding gas is required to control the atmosphere inside the furnace. This environment is protected from oxygen by the use of argon. At room temperature, argon is chemically inert under most conditions; thus, low thermal conductivity forms no confirmed stable compounds [24].Pure magnesium ingots were preheated to 400°C for an hour. Then, the metal was heated to 750°C. The reinforcement material was added to the molten metal. The holding time between the matrix and the reinforcement is measured as an important factor in the processing of composites. When the holding time was 10 minutes, the particles were distributed uniformly in the matrix. The melt was then solidified in permanent moulds in the form of a 25 mm rod with 250 mm in height. Then, using EDM, the samples were machined to get the required dimensions for conducting various experiments.

2.3 Energy Dispersive X-ray (EDX) spectroscopy and Scanning Electron Microscopy (SEM)

Samples were machined from extruded bars, then polished and characterized for their microstructures. The samples were polished with an automatic polisher until mirror-like surfaces were obtained. The EDX spectroscopy is used to identify the constituents of chemical elements in the prepared composites. The samples machined by EDM were subjected to the EDX analysis for identifying the presence of particles and their peak values. The phase analysis was carried out at a speed of 3 degrees/minute with a range of 0-100 degrees. As the intensities agree with the theoretical values, the increase in the peak areas gives the information about the kinetics of the reaction process. The X-ray diffraction data were obtained using a Rigaku D/Max-B X-ray diffractometer with the Bragg–Brentano parafocusing geometry, a diffracted beam monochromator, and a conventional copper target X-ray tube set to 40 kV and 30 mA. The EDX images of the reinforcements (2.5%, 5%, and 7.5%) with magnesium matrices are presented in Fig.2 (a-c).

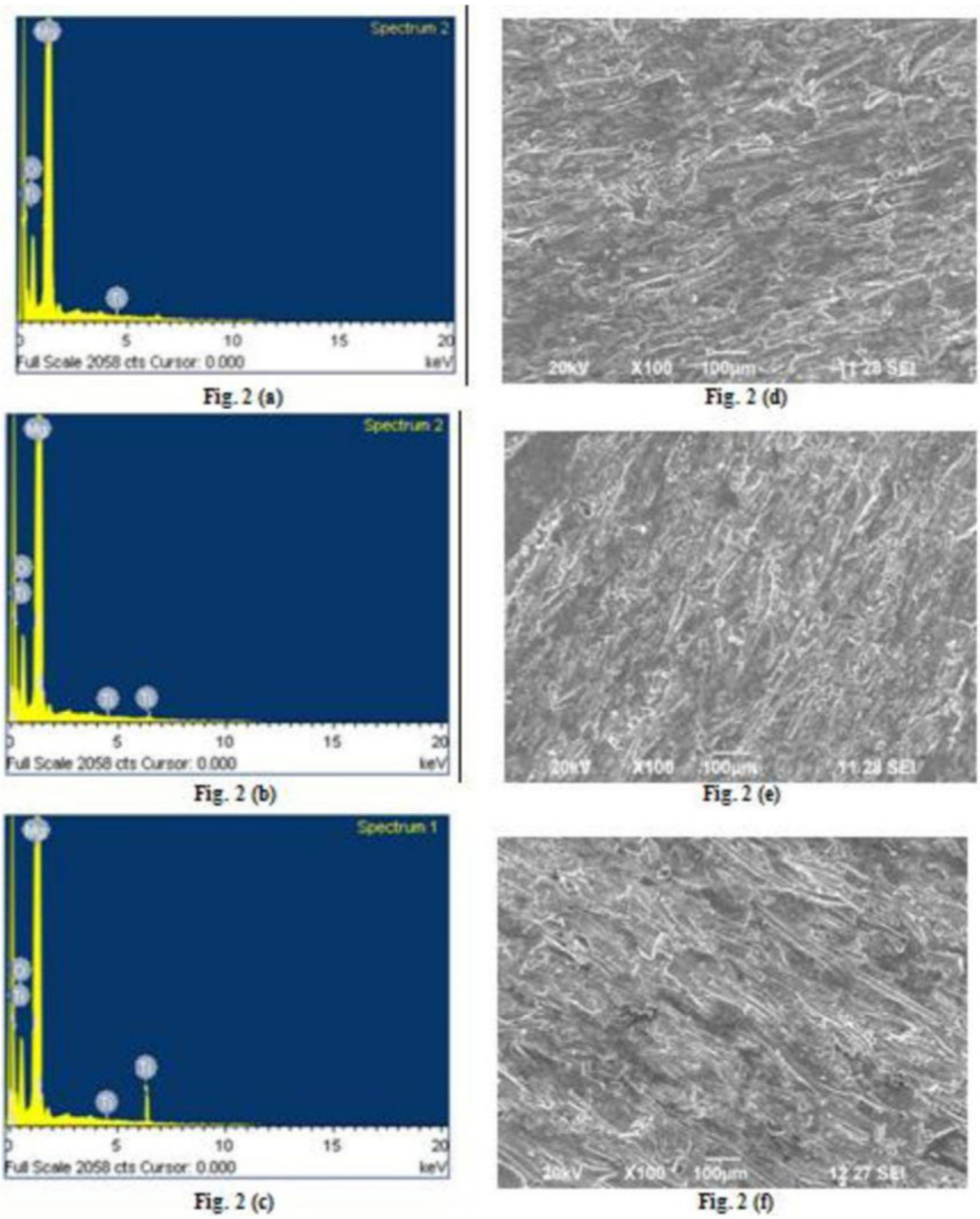


Fig. 2 (a-c) EDX patterns of Mg+2.5% TiO_2 , Mg+5% TiO_2 , Mg+7.5% TiO_2.
(d-f) SEM images of Mg+2.5% TiO_2, Mg+5% TiO_2 , Mg+7.5% TiO_2 at 100X magnification

It is evident that TiO_2 is fully formed and a large quantity of molten magnesium infiltrates through the aperture gap of the particulate. One can clearly see fin these images that magnesium, titanium, and oxygen are present in the prepared samples. The topography of the upper surfaces is analysed by means of a scanning electron microscope. The morphology of the developed composites is analysed using SEM and the images are presented in Fig. 2 (d-f). Uniform distribution of the particles in the magnesium matrix is observed. There are no micropores or cracks in the surface due to the effective stirring process. In addition, there is no accumulation of particles.

2.4 Dry sliding wear test

Dry sliding wear tests were conducted using a computerized pin-on-disc tester. The sectioning of pin samples of size 10 mm x 10 mm x 12 mm was performed by EDM. The contact surfaces were prepared by grinding (600-grit silicon carbide paper was used) and were then cleaned with alcohol. A pin holder loaded the stationary pins vertically onto a rotating AISI-O1 tool steel disc, which had been oil-hardened to 63 HRC. All experiments were conducted in air with the temperature and the relative humidity maintained at 20-25°C and 55– 67%, respectively. Four normal loads (10, 15, 20, and 25 N) were applied using dead weights, and three sliding velocities (1, 1.5, and 2.0 m/s) were selected. The pin-on-disc equipment and wear samples are presented in Fig. 3 (a,b).

(a) (b)

Fig. 3 a) Pin-on-Disc machine b) Wear samples with various contents of TiO_2

For the 1 m/s sliding condition, the test was carried out for 10 min at a speed of 320 rpm. Similarly, for the sliding velocities of 1.5 m/s and 2 m/s, the test was carried out 10 min at the sliding velocities of 480 rpm and 640 rpm, respectively. Prior to each test, the disc was ground with 600-grit SiC paper for a few minutes to remove the accumulated debris on the wear track, and then it was cleaned with alcohol. At the end of each test, the pins were carefully cleaned with alcohol and weighed on a sensitive electronic balance with an accuracy of ±0.1 mg to determine the weight loss. The difference between the weight before and after the test was calculated as wear loss. The worn pin surfaces and the collected wear debris were examined and analysed using SEM.

2.5 Surface roughness test

The interaction of the developed composites with the environment is greatly influenced by the surface roughness experiments. Therefore, the surface roughness of the developed Mg-TiO_2 composites was measured before and after the wear test by a Mitutoyo surftest SJ -210 portable surface roughness tester and the results were discussed in detail in section 3.4. It was investigated whether the surface texture greatly influences the functioning of machined parts.

It was determined that properties like corrosion resistance, wear resistance, and fatigue resistance greatly depend upon the surface texture. A surface roughness tester with a 2.4 inch colour graphic back-lit LCD provided excellent readability and display of worn-out samples.

3. Results and discussions

3.1 Effect of the content of reinforcements on wear loss

Samples prepared with different weight percentages of reinforcements are pressed against the disc to determine the wear loss of each sample. The values of wear losses are plotted in Fig 4.a. Each sample is tested under a particular load for 10 minutes. Dead weights ranging from 10 N to 25 N are applied. The graph shows that the wear loss of samples increases with an increase in loads. The 25 N load incurs a greater amount of wear loss than other loads. A comparison of wear losses with the percentage of reinforcements in the matrix shows that the pure magnesium samples exhibit a greater amount of wear loss. The addition of particles produced better adhesion between the matrix and the particles, and this in turn resulted in improved resistance to delamination. Thus, the addition of TiO_2 particles improves the wear resistance of the magnesium matrix in Mg-TiO_2 composites. Also, the wear loss curves obtained for all the samples develop in a non-linear manner [25-27]. Therefore, it is inferred that the wear loss does not vary proportionally with respect to a change in load.

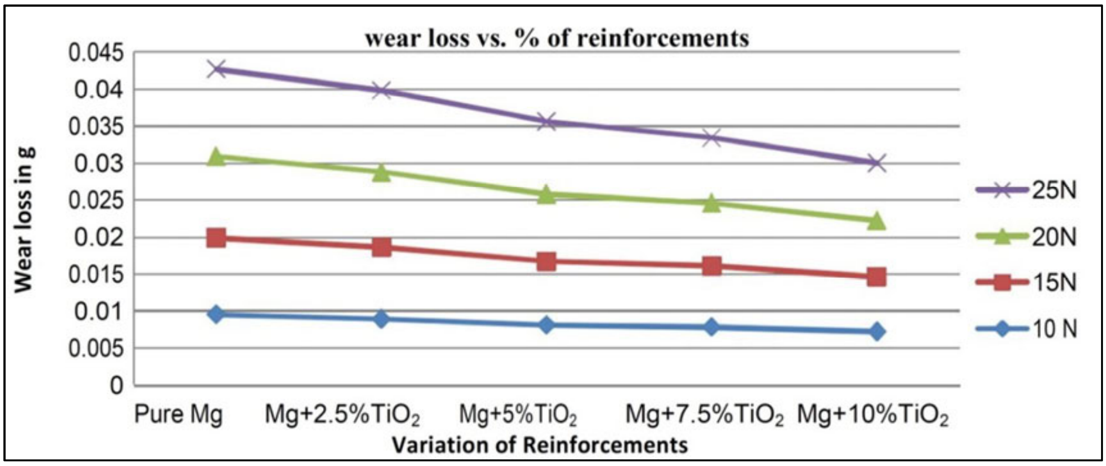


Fig. 4.a) Wear loss with the variation of reinforcements

3.2 Effect of loads on the wear loss

It is evident that wear losses will be increasing with an increase in load. The results confirmed that principle shown in Fig 4.a). The variation in load helps us to understand the behaviour of a candidate material during the metal-to-metal contact even under higher loads. It is noticed that while the percentage of reinforcement increases, the wear loss of the sample decreases. The increase in wear loss of Mg-TiO_2 composites at high loads may be caused by the brittle nature of the reinforcement particles. The presence of ceramic reinforcements increases the wear resistance of matrix materials at a lower range of applied loads. The composite generally exhibits better wear resistance under lower loads due to its superior load-bearing capacity and its ability to maintain a stable oxide film which protects it from metal-to-metal contact during sliding.

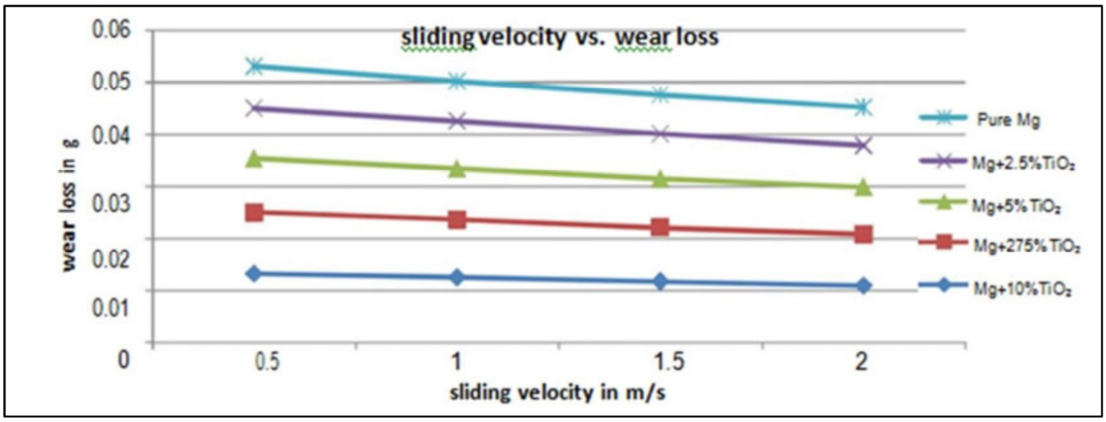


Fig 4.b) Wear loss with the variation in sliding velocity

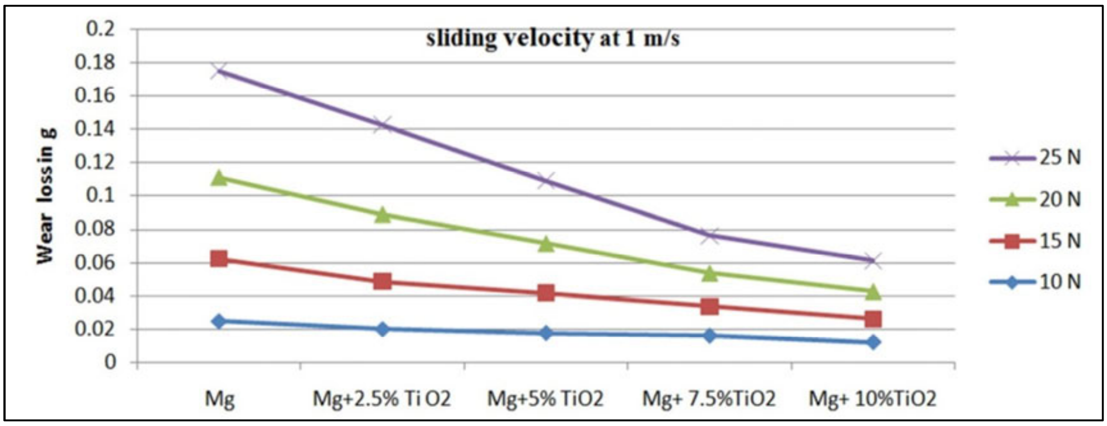


Fig 4.c) Wear loss for different samples at 1 m/s

3.3 Effect of sliding velocity on wear loss

The sliding velocity is kept constant at 1, 1.5, and 2 m/sand the loads vary from 10 N to 25 N; wear losses under these conditions were measured and the results are shown in Fig. 4 (b-e). The interpretation of the results indicates that the wear loss decreases with an increase in the sliding velocity regardless of the quantum of reinforcements. When the sliding velocity increases, a thinner and less adherent lubricant layer peels off from the surface of the composite and in turn forms an oxide layer which prevents the test sample from further loss. The increase in the sliding velocity decreases the contact time between the pin and the disc, resulting in a decreasing wear loss.

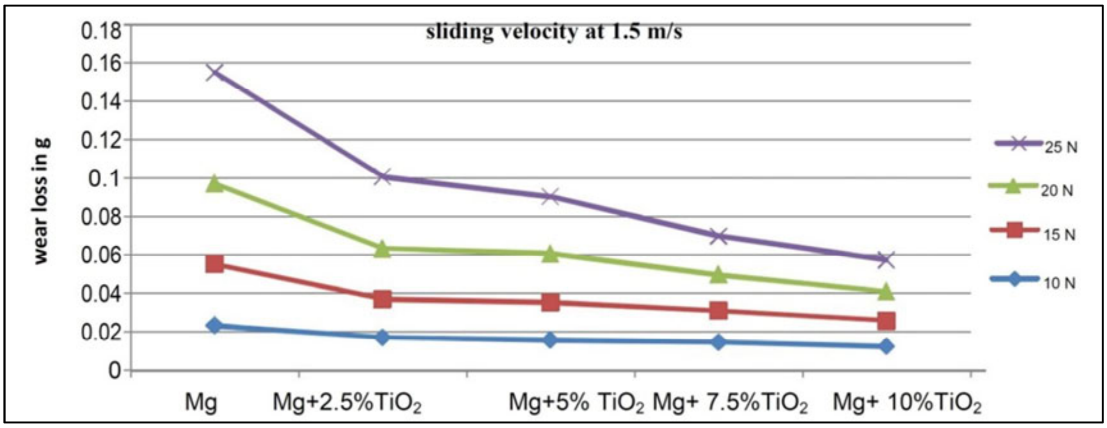


Fig 4.d) Wear loss for different samples at 1.5 m/s

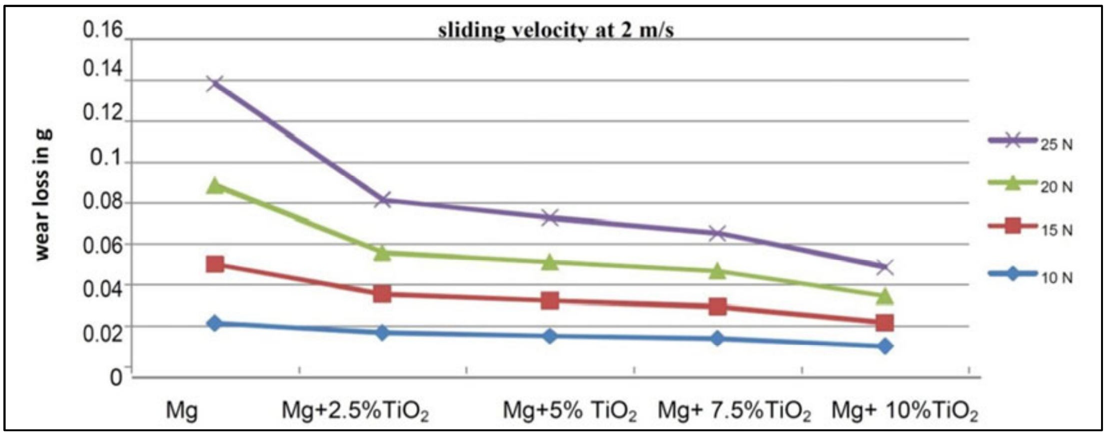


Fig 4.e) Wear loss for different samples at 2 m/s

The variation in the sliding distance will increase the time of contact of continuous sliding over the hard surface, which may cause an increase in temperature at the interface. This increase in temperature may cause the samples to soften, which results in an increased wear loss. The reduction in wear loss of the samples with a significant increase in the amount of TiO_2 particles at higher sliding distances may be a result of the wear resistance produced by the effect of magnesium interfacing with TiO_2 reinforcements.

3.4 Measurement of surface roughness (Rp)

The measured values of surface roughness (R_p) of the prepared samples before and after the wear test are presented in Fig. 5. Since the maximum applied load of 25 N produced the greater wear losses to the pin samples, corresponding samples were taken for the surface roughness test. It is evident that the surface roughness values decrease with the increase in the amount of TiO_2 reinforcement particles added to the matrix.

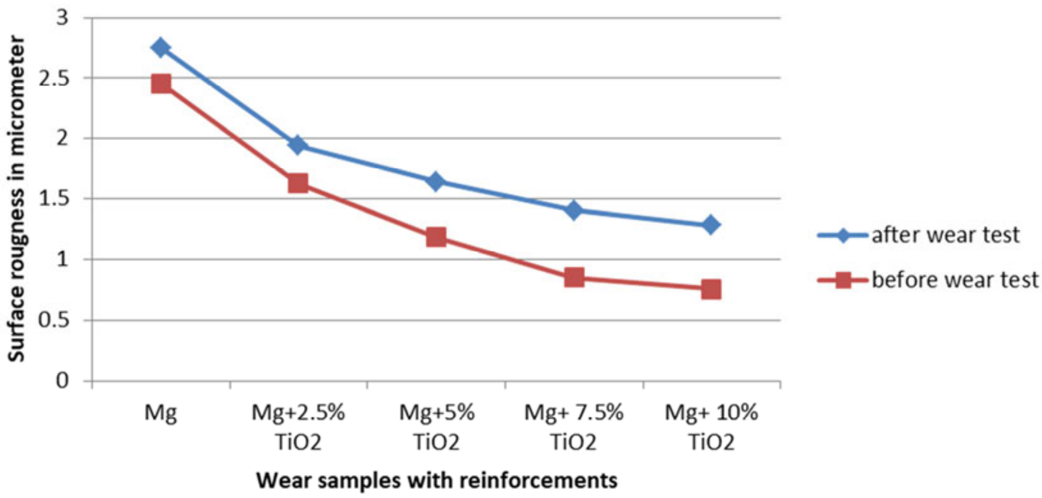


Fig. 5 Surface roughness values for different samples

4. Conclusions

The main conclusions are as follows:

1. Magnesium-based TiO_2 composites which were successfully synthesized by the argon-controlled vacuum stir casting method exhibited minimal porosity.
2. EDX patterns proved the presence of constituent chemical elements in the composites according to the variation in reinforcements.

3. SEM analysis ensured the uniform distribution of the particles throughout the composites.
4. Pin-on-disc dry sliding wear tests were carried out by holding Mg-based composite pins against a rotating steel disc under loads of 10 to 25 N and at the 1–2 m/s range of sliding velocities.
5. The composites exhibit slightly superior wear resistance under the lower load regardless of the reinforcement. The dominant wear mechanism under the lower load of 10N is oxidation.
6. Wear resistance is improved in accordance with the addition of particles.
7. Wear loss in composites increased with an increase in load.
8. At a lower sliding velocity, wear loss is higher than at higher velocities.

REFERENCES

[1] Zhong, X.L.; Wong,W.L.E.; Gupta. M. Enhancing strength and ductility of magnesium by integrating it with aluminum nanoparticles, ActaMaterialia, 55, 6338–6344 (2007). https://doi.org/10.1016/j.actamat.2007.07.039

[2] Karthikeyan, G.; Jinu, G.R.; Dry Sliding Wear Behaviour of Stir Cast LM25/ZrO_2 Metal matrix composites, Transaction of FAMENA, 39 (4), 89-98 (2015).

[3] Falcon-Franco, L.; Bedolla-Becerril, E.; Lemus-Ruiz, J.; Gonzalez-Rodríguez, J.G.; Guardian, R.; Rosales, I. Wear performance of TiC as reinforcement of a magnesium alloy matrix composite, Composites: Part B, 42, 275–279 (2011). https://doi.org/10.1016/j.compositesb.2010.11.012

[4] An, J.; Li, R.G.; Lu, Y.; Chen, C.M.; .Xu, Y.; Chen, X.; Wan, L.M.; Dry sliding wear behavior of magnesium alloys, Wear, 265, 97–104 (2008). https://doi.org/10.1016/j.wear.2007.08.021

[5] Aitao Tang, Fusheng Pan, Mingbo Y, Renju Cheng, Mechanical Properties and Microstructure of Magnesium-Aluminum Based Alloys Containing Strontium, Materials Transactions, 49, 6 , (2008) pp. 1203 - 1211.

[6] Gupta, M.; Lai, M.O.; Sarvanaranganathan, D. Synthesis, microstructure and properties characterization of disintegrated melt deposited Mg/SiC composites. J. Mater. Sci., 35, 2155–2165 (2000). https://doi.org/10.1023/A:1004706321731

[7] Uganda, S.; Gupta, M.; Sinha, S.K. Enhancing strength and ductility of Mg/SiC composites using recrystallization heat treatment. Compos. Struct.,72, 266–272 (2006). https://doi.org/10.1016/j.compstruct.2004.11.010

[8] Sankaranarayanan Seetharaman, Jayalakshmi Subramanian, Manoj Gupta and AbdelmagidHamouda, Influence of Micron-Ti and Nano-Cu Additions on the Microstructure and Mechanical Properties of Pure Magnesium, Metals, 2, 274-291 (2012). https://doi.org/10.3390/met2030274

[9] Sozhamannan, G.G.; Balasivanandha Prabu, S.; Venkatagalapathy, V.S.K. Effect of Processing Parameters on Metal Matrix Composites: Stir Casting Process, Journal of Surface Engineered Materials and Advanced Technology, 2, 11-15 (2012). https://doi.org/10.4236/jsemat.2012.21002

[10] Samal, B.P.; Panigrahi, S.C.; Sarangi, B. Use of Modified Stir Casting Technique to Produce Metal Matrix Composites, International Journal of Engineering and Technical Research (IJETR), 1, 9, pp 63-65 (2013).

[11] Vettivel, S.C.; Selvakumar, N.; Narayanasamy, R.; Leema, N. Numerical modeling, prediction of Cu-W nano powder composite in dry sliding wear condition using response surface methodology, Material and Design, 50, 977-996, (2013). https://doi.org/10.1016/j.matdes.2013.03.072

[12] Ganesh Kumar, Manoj Gupta, Low volume fraction nano-titanium particulates for improving the mechanical response of pure magnesium, Journal of alloys and compounds, 593, 576-583 (2014).

[13] Chen, Q.; Shu, D.; Zhao, Z.; Wang, Y.; Yuan, B.; Microstructure development and tensile mechanical properties of Mg–Zn–RE–Zr magnesium alloy. Material and Design, 40, 488–96 (2012). https://doi.org/10.1016/j.matdes.2012.03.059

[14] Tun, K.; Jayaramanavar, P.; Nguyen, Q.; Chan, J.; Kwok, R.; Gupta, M. Investigation into tensile and compressive responses of Mg–ZnO composites. Material Science Technology, 28, 582–588 (2012). https://doi.org/10.1179/1743284711Y.0000000108

[15] Meenashisundaram, G. K; Gupta, M. Emerging Environment Friendly, Magnesium-Based Composite Technology for Present and Future Generations, The Minerals, Metals & Materials Society, 2016.

[16] Sanjay Kumar Thakur, Brij Kumar Dhindaw, The influence of interfacial characteristics between SiCp and Mg/Al metal matrix on wear, coefficient of friction and microhardness, Wear, 247, 191–201 (2011). https://doi.org/10.1016/S0043-1648(00)00536-6

[17] Lim, C.Y.H.; Leo, D.K.; Ang, J.J.S.; Gupta, M. Wear of magnesium composites reinforced with nano-sized alumina particulates, Wear, 259, 620–625 (2005). https://doi.org/10.1016/j.wear.2005.02.006

[18] Habibnejad-Korayem, M.; Mahmudi, R.; Ghasemi, H.M.; Poole, W.J. Tribologicalbehavior of pure Mg and AZ31 magnesium alloy strengthened by Al2O3 nano-particles, Wear 268, 405–412 (2010). https://doi.org/10.1016/j.wear.2009.08.031

[19] Wang, X.J.; Wu, K.; Zhang, H.F.; Huang, W.X.; Chang, H.; Gan, W.M.; Zheng, M.Y.; Peng, D.L. Effect of hot extrusion on the microstructure of a particulate reinforced magnesium matrix composite, Materials Science and Engineering A, 465, 78–84 (2007). https://doi.org/10.1016/j.msea.2007.03.077

[20] Mordike, B.L.; Ebert, T.; Magnesium: Properties — applications — potential, Materials Science and Engineering: A, 302,1, 37–45 (2001). https://doi.org/10.1016/S0921-5093(00)01351-4

[21] Hassan, S.F.; Ho, K.F.; and Gupta, M. Synthesis of High-Performance Magnesium/Cu, Ni, Ti Composites, Journal of material technology,17, 224-228 (2002).

[22] Wong, W.L.E.; Gupta, M. Development of Mg-Cu nanocomposites using microwave assisted rapid sintering, Composites Science and Technology, 67, 1541–1552 (2007). https://doi.org/10.1016/j.compscitech.2006.07.015

[23] Tun, K.S.; Gupta, M. Improving mechanical properties of magnesium using nano-yttria reinforcement and microwave assisted powder metallurgy method. Composites Science and Technology, 67, 2657–2664 (2007). https://doi.org/10.1016/j.compscitech.2007.03.006

[24] Moll, F.; Kainer, K.U.; Magnesium: Alloys and Technology (Weinheim: Wiley), 197–217 (2004).

[25] Nuckols M.L.; Giblo J.; Wood-Putnam JL ., Thermal Characteristics of Diving Garments When Using Argon as a Suit Inflation Gas, Institute of Electrical and Electronics Engineers, Inc. (IEEE),(2009).

[26] Tjong, S.C.; Lau, K.C.; Abrasive wear behavior of TiB2 particle-reinforced copper matrix composites. Mater Sci. Eng A, 282,183–186 (2000). https://doi.org/10.1016/S0921-5093(99)00752-2

[27] Mandal, A.; Chakraborty, M.; Murty B.S. Effect of TiB2 particles on sliding wear behavior of Al–4Cualloy. Wear, 262,160–166 (2007). https://doi.org/10.1016/j.wear.2006.04.003

Submitted: 07.02.2017

Accepted: 26.6.2017

Autchipillai Lakshmanan Pillai
Faculty Head,
alakme2000@yahoo.co.in
Department of Mechanical Engineering, Cape Institute of Technology,
Kanyakumari, Tamilnadu, India

Gowthami Thankachi Raghuvaran Jinu,
Assistant Professor,
gr_jinu1980@yahoo.com
Department of Mechanical Engineering,
University College of Engineering,
Nagercoil-629004, Tamilnadu, India

Govindan Karthikeyan,
Assistant Professor,
p_gkarthikeyan@yahoo.co.in
Department of Mechanical Engineering,
Regional Campus of Anna University,
Tirunelveli Region, Tirunelveli, India